II.D.8 Montana Palladium Research Initiative – Palladium-Based Membrane on a Porous Stainless Steel Substrate*

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*Congressionally directed project

Objectives

- Design membrane substrate with emphasis on strength, surface finish, and consistent pore size.
- Incorporate an intermediate metallic oxide layer to buffer the palladium membrane from contamination and to provide a smooth surface for the membrane plating.
- Develop full density palladium membrane layer that is thin, with high flux rate, high impurity tolerance, durable in operating conditions, and a strong adherence to the substrate.
- Design and manufacture a permeation measurement system.
- Safely perform module/membrane testing.

Technical Barriers

This project addresses the following technical barriers from the Hydrogen Separations Section of the Hydrogen, Fuel Cells and Infrastructure Technologies Program Multi-Year Research, Development and Demonstration Plan (April 27, 2007):

(K) Membrane Durability
(L) Impurities
(M) Membrane Defects
(P) Flux
(Q) Testing and Analysis
(R) Cost

Technical Targets

Fabrication and testing will be compared against the DOE Technical Targets (see Table 1).

TABLE 1. Technical Targets: Dense Metallic Membranes for Hydrogen Separation and Purification

Performance Criteria	Units	2010 Target	2015 Target	CAMP 2007 Status
Flux Rate	scfh/ft^2	250	300	In the design phase
Module Cost (including membrane material)	\$/ft^2 of membrane	1,000	<500	
Durability	hr	26,280	>43,800	
Operating Capability	psi	400	400-600	
Hydrogen Recovery	%	>80	>90	
Hydrogen Quality	% of total (dry) gas	99.9	>99.99	

Accomplishments

- Performed free form fabrication of the metallic substrate.
- A test piece using 30-mm stainless steel powders has been successfully fabricated. Two 3D computer aided design (CAD) models of potential substrates have been completed.
- Electroless plating has been determined to be appropriate for fabricating the palladium membrane.
- A preliminary design for the module testing apparatus has been developed.
- It has been determined the Montana Tech's scanning electron microscope, equipped with a back-scatter detector and mineral liberation analysis (MLA) software can be used for composition identification.
- A hydrogen safety training class has been developed.

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Introduction

The design of the porous stainless steel substrate is critical to the durability of the palladium membrane system. The objectives for this substrate development include ability to withstand elevated operating temperatures and pressures, structural stability to endure operating stresses and strains, chemically resistant, sufficient porosity, and include appropriate connection mechanisms (flange, pipe thread, etc.).

Approach

Hydrogen is uniquely soluble in bulk palladium; therefore a structurally sound palladium-based membrane can separate highly pure hydrogen. Due to the excessive cost of ultra-high purity hydrogen in compressed gas form, there has been a great interest in fuel processing technologies such as steam reforming of methane where liquid fuel is reformed into H_2 and CO_2. A thin palladium-based membrane can play an important role in this fuel processing technology. The thinner the membrane the greater the hydrogen flux across the membrane and the smaller the area required. This membrane must be sufficiently thick enough to withstand pinhole failures due to the elevated pressures and temperatures of the reformer. The Center for Advanced Mineral and Metallurgical Processing, located on the campus of Montana Tech, will investigate plating a palladium membrane onto a porous stainless steel substrate. This substrate will be manufactured onsite with stainless steel powders and a ProMetal R2 3D printing machine. The palladium will be plated onto the porous tube filter through the use of an electroless-plating bath. In addition, a durable, thin palladium-based membrane, that will maintain its integrity within a catalyst bed, will be very attractive to the fuel cell industry and other potential hydrogen consumers.

It has been determined that Montana Tech/CAMP has in-house expertise for welding porous stainless steel onto machined stainless steel necessary for attachment. The Montana Tech Rocky Mountain Agile Virtual Enterprise (RAVE) Center contains several computer numerical control (CNC) mills and various welding machines that are appropriate for the necessary fabrication.

ProMetal R2 3D Printing Process

- The fabrication process begins with a 3D CAD image of the component.
- Upon completion, the file is saved as a ".stl" file. This format saves a 3D file as a series (or layers) of 2D files and is downloaded to the ProMetal R2 3D printing machine and the printing procedure begins.
- The ProMetal R2 feed chamber indexes upward and the build chamber indexes downward (see Figure 1).

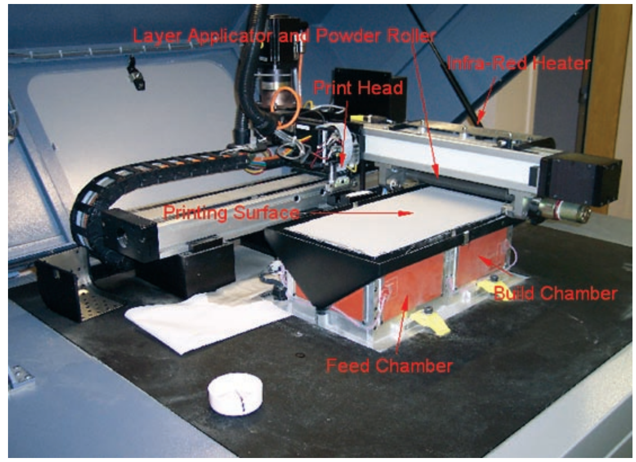

FIGURE 1. ProMetal R2 Components

- The layer applicator and powder roller pushes powder from the feed chamber to the build chamber, creating a relatively flat surface of metal powders.
- The print head deposits binder droplets at sites dictated by the ".stl" file.
- The printed layer is dried by the heater (infrared lamp).
- Steps 3 through 4 are repeated until each layer in the ".stl" file have been printed.
- The component is then subjected to two thermal processes: low temperature binder curing and sintering.

Results

- Successful implementation and training for ProMetal R2 – 3D printing machine (see Figure 2).
- Conceptual design of substrate and permeation measurement system (see Figure 3).

Conclusions and Future Directions

- Procure equipment: vacuum sintering furnace and particle shape and size analyzer.
- Complete evaluation of metal powders for free form fabrication.
- Complete development of substrate models.
- Complete development of the sintering schedule.
- Fabricate the substrate models.
- Complete the evaluation of the palladium and palladium alloys for the membrane.

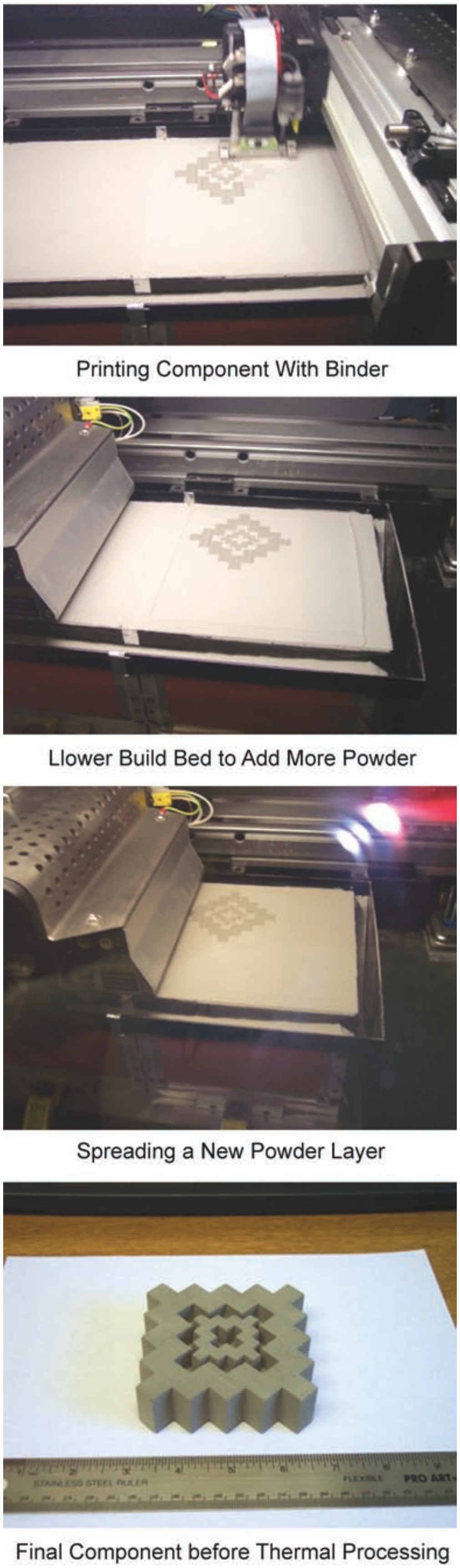

FIGURE 2. Successful 3D Printing of Test Piece

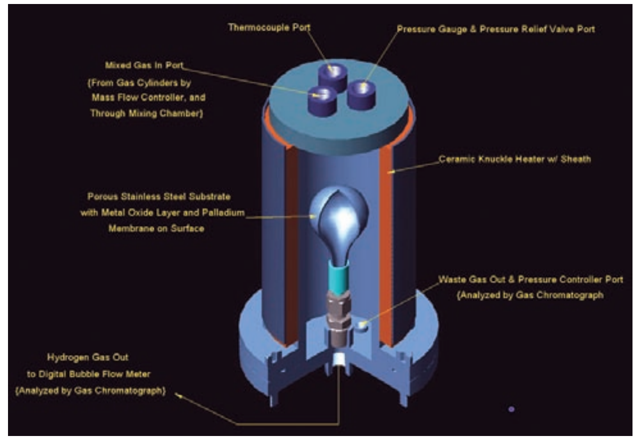

FIGURE 3. Conceptual Design of Substrate and Permeation Measurement System

FY 2007 Publications/Presentations

1. 2007 DOE Hydrogen Program Review – Poster Session. Arlington, VA, May 15, 2007.